## PATENT ABSTRACTS OF JAPAN

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## (54) COMPOSITION FOR ORAL CAVITY

## (57) Abstract:

PROBLEM TO BE SOLVED: To obtain a composition for oral cavity having high cleaning effect, low polishing property and high commercial value.

SOLUTION: This composition for oral cavity contains 0.2-20 wt.% crystalline cellulose powder and calcium hydrogen phosphate. nonhydrate having crystallite with 300-3,500 & angst; diameter (average value) measured by X ray diffraction method. The compounding amount of calcium hydrogen phosphate.nonhydrate is preferably 5-60% and sufficient cleaning force is not obtained when the calcium hydrogenphosphate content is <5% and the polishing force becomes too high when the content exceeds 60%. More preferable calcium hydrogenphosphate content is 10-50%.

#### **CLAIMS**

#### [Claim(s)]

[Claim 1]A constituent for the mouths containing calcium hydrogen phosphate and anhydrate which has the microcrystal whose average value which measured crystal cellulose powder with 0.2 to 20 % of the weight and an X-ray diffraction method is 300-3500A.

#### **DETAILED DESCRIPTION**

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the constituent for the mouths which has the outstanding cleaning effect.

[0002]

[Description of the Prior Art]Conventionally, it mainly depends for the cleaning effect of constituents for the mouths, such as toothbrushing, on the grinding force of abrasive soap, such as inorganic powder.

Since the cleaning effect of this constituent for the mouths was in proportionality mostly with the grinding force of abrasive soap, when a cleaning effect was heightened, it needed to increase the loadings of abrasive soap.

However, when the loadings of abrasive soap were increased, while the cleaning effect became high, it had a possibility of having deleted a tooth according to the grinding force, and causing damage. Then, grinding force was not raised but development of the high constituent for the mouths of a cleaning effect was desired.

[0003]On the other hand, it is publicly known to use crystal cellulose powder for the constituent for the mouths, for example, it is applied to the aqueous gel-like constituent of fluoride content for the purpose of the paint of fluoride (JP,61-192750,A). The proposal (JP,60-188309,A) which blended calcium hydrogen phosphate and anhydrate whose average value of microcrystal is 300-3500A for the purpose of mild polish nature exists. However, cleaning effect sufficient in these things was not acquired. [0004]

[The means for solving a technical problem and an embodiment of the invention] This invention persons did the knowledge of the above-mentioned purpose being attained by using together calcium hydrogen phosphate and anhydrate whose average value of crystal cellulose powder and microcrystal is 300-3500A, as a result of repeating examination wholeheartedly, in order to obtain the constituent for the mouths which gives sufficient cleaning effect without raising grinding force.

[0005]Per this invention and also the crystalline cellulose which will be used for this invention if it explains in full detail hereafter, Acidolysis or the cellulose microcrystal aggregate which is produced by carrying out alkali hydrolysis and which has a fixed degree of polymerization substantially is said for the alpha cellulose obtained from fibrous vegetation as pulp, As [ define / as Mr. sea devil PACHISUTA's report indicated to industrial and engineering chemistry, the 42nd volume (1950), and the 502-507th page / for example, ] If it is preferred that it is 50-200 micrometers and it is smaller than 50 micrometers, sufficient cleaning power will not be obtained, but if the mean particle diameter in the dryness of crystalline cellulose is larger than 200 micrometers, it will give foreign body sensation at the time of use, and the case where a using feeling is worsened produces it. More desirable mean particle diameter is 70-150 micrometers.

[0006]The loadings of the above-mentioned crystalline cellulose are 0.2 to 20% of the whole constituent (it is below the same% of the weight), if cleaning power with loadings sufficient at less than 0.2% is not obtained but it exceeds 20%, foreign body sensation will be given at the time of use, and a using feeling will be worsened. It is 1 to 5% still more preferably 0.5 to 10% preferably.

[0007]On the other hand, a size of microcrystal which measured calcium hydrogen phosphate and anhydrate used for this invention with an X-ray diffraction method is 300-3500A. Sufficient cleaning power is not obtained with it being less than 300A, but if it exceeds 3500 A, grinding force will become high too much. It is 500-3000A preferably, and is 1000-2000A still more preferably. It is 3800-4300A, and if cleaning power is made high even if it uses together with crystal cellulose powder average value of a size of microcrystal which measured calcium hydrogen phosphate and anhydrate currently used conventionally with an X-ray diffraction method, grinding force becomes high and it is not desirable. [ of grinding force ]

[0008]Cleaning power in which it is [ that it is 5 to 60% ] preferred it as for loadings of

calcium hydrogen phosphate and anhydrate, and it is sufficient at less than 5% is not obtained, but if it exceeds 60%, a case where grinding force becomes high too much will arise. It is 10 to 50% more preferably.

[0009]Although a constituent for the mouths concerning this invention can be prepared for tooth paste, liquefied toothbrushing, etc., in this case, as other ingredients, a proper ingredient by which normal use is carried out can be blended, and abrasive soap other than the above, a binder, a viscous agent, a surface-active agent, a sweetening agent, perfume, an antiseptic, various active principles, etc. may be blended.
[0010]As abrasive soap, for example, calcium hydrogen phosphate and 2 hydrate, tribasic calcium phosphate, Calcium carbonate, calcium pyrophosphate, aluminium hydroxide, a silicic acid anhydride, One sort, such as aluminum silicate, insoluble sodium metaphosphate, the 3rd magnesium phosphate, magnesium carbonate, calcium sulfate, bentonite, zirconium silicate, poly methyl methacrylate, and other synthetic resins, or two sorts or more can be blended in the range which does not spoil an effect of this invention.
[0011]As a binder, a carrageenan, carboxymethylcellulose sodium, Cellulosics, such as

[0011]As a binder, a carrageenan, carboxymethylcellulose sodium, Cellulosics, such as methyl cellulose and hydroxyethyl cellulose, Alginic acid derivatives, such as sodium alginate and propylene glycol alginate, xanthan gum and jouet -- one sort, such as inorganic binders, such as synthetic binders, such as gums, such as run gum, tragacanth gum, and karaya gum, polyvinyl alcohol, sodium polyacrylate, and a carboxyvinyl polymer, silica gel, veegum, and RAPONAITO, or two sorts or more can be blended. [0012]As a moisturizer, one sort of polyhydric alcohol, such as glycerin, sorbitol, propylene glycol, a polyethylene glycol, xylitol, maltitol, and a RAKUSHI toll, or two sorts or more can be blended.

[0013] As a surface-active agent, ampholytic surface active agents, such as nonionic surfactants, such as anionic surface-active agents, such as sodium lauryl sulfate, lauric acid decaglyceryl, and myristic acid diethanolamide, and a betaine series, can be blended. [0014] As a perfume component, menthol, anethole, carvone, eugenol, Limonene, n-decyl alcohol, citronellol, alpha-terpineol, Cineol, linalool, ethyl linalool, WANIRIN, Timor, It is independent, or perfume, such as peppermint oil, spearmint oil, wintergreen oil, clove oil, and eucalyptus oil, is combined, and can be blended, and also sweetening agents, such as saccharin sodium, stevioside, glycyrrhizin, perilla rutin, and thaumatin, can be blended.

[0015]As an active principle, fluorides, such as sodium fluoride and sodium monofluorophosphate, A dextranase, mutanase, a lysozyme, amylase, protease, Enzymes, such as lytic enzyme, tranexamic acid, epsilon aminocaproic acid, One sort of active principles, such as glycyrrhetinic acid, bisabolol, chlorophyll, sodium chloride, a water-soluble inorganic-phosphoric-acid compound, triclosan, cetyl pyridinium chloride, a benzalkonium chloride, and benzethonium chloride, or two sorts or more can be blended. [0016]

[Effect of the Invention]By having used together crystal cellulose powder, and calcium hydrogen phosphate and anhydrate which have the microcrystal whose average value measured by the X-ray diffraction method is 300-3500A, a cleaning effect is high, the constituent for the mouths of this invention gives the constituent of low polish nature, and its commodity value is high.

[0017]

[Example] Hereafter, although the example of an experiment and an example explain the

effect of this invention concretely, this invention is not restricted to the following example. Each % in each example is weight %. The microcrystal of calcium hydrogen phosphate and anhydrate was measured by the following method. [0018] The X diffraction of the measuring method granular material of microcrystal was performed, and the size of microcrystal was quantitatively expressed for the crystallinity of the granular material as an index from blow donning of the peak. Here, X line source was measured using Cu-K alpha rays, analyzed the data of the X diffraction about the main peaks which do not have a lap using formula D=K lambda/beta costheta of Scherrer, and asked for the size of average microcrystal. In this case, the average was taken as main peaks about 2theta=53.1 degree, 49.3 degrees, 47.3 degrees, 36.1 degrees, 32.9 degrees, 32.6 degrees, 31.1 degrees, 30.25 degrees, 28.65 degrees, and 13.15 degrees. The breadth (the [rad] alpha-aluminum 2O<sub>3</sub> granular material was used at 1100 \*\* on the basis of what was calcinated for 24 hours) of a diffraction line purely based on [ based on / based on the size (A) of microcrystal in D / measurement X-rays wavelength (A) in lambda | the size of microcrystal in beta, and K A form factor (constant = referred to as 0.9), theta was taken as the bragg angle of the diffraction line. beta is the value which deducted the half breadth given with the very good crystalline substance under the same conditions from the half breadth for which it asked experimentally. [0019][The example 1 of an experiment] The kind shown in Table 1 and the following dentifrices containing the ingredient of concentration were prepared, and the following method estimated the cleaning effect. A result is shown in Table 1.

<Dentifrices presentation> cellulose powder Quantity shown in Table 1 Crystalline cellulose Quantity shown in Table 1 Calcium hydrogen phosphate and anhydrate Ouantity shown in Table 1 (the size of microcrystal: 1660A) Sodium lauryl sulfate 1.5% Carrageenan 1 % sorbitol 25 % perfume 1 % purified water \*\*\*\*\* 100 %[0021]Collect cleaning power appraisal method TABAKOYANI by the usual method, and make this into solution form and it is uniformly applied on a tile, After carrying out stoving, this was set to the polishing vessel, 15 g of toothbrushing was brushed 2000 times in 200 g of load using the liquid uniformly distributed in the glycerin 25g 85%, and the extraction ratio of TABAKOYANI of a tile was evaluated after brushing with the naked eye. As a brush, the thing of M was used by the home article descriptive label method with nylon construction material the thickness of 8 mils (about 0.2 mm) of the number of hair-bundles, and 44 hair, and 12 mm [woolen] in length. Valuation-basis marks 1: TABAKOYANI extraction ratio 0 to 20%2: TABAKOYANI extraction ratio 21 to 40%3: TABAKOYANI extraction ratio 41-60%4: TABAKOYANI extraction ratio 61-80%5: TABAKOYANI extraction ratio 81 to 100% [0022] [Table 1]

-8.0	サンブルNo.									
成分	1	2	3	4	5	6	7	8	9	10
セルロース粉末	10	_	-	2	-	_	_	_	_	-
結晶セルロース (平均粒径 120 μ m)	_	10	-		0.1	5	1	10	15	0.5
リン酸水素カルシウム・ 無水和物	-	1	5	7	5	10	25	10	30	10
<b>濟揚力評点</b>	1	1	2	2	2	4	4.5	4	5	4
	比較品					本発明品				

[0023]From the result of Table 1, when crystalline cellulose, and calcium hydrogen phosphate and anhydrate were used together according to this invention, it was checked that a high cleaning effect is demonstrated.

[0024][The example 2 of an experiment] Using calcium hydrogen phosphate and anhydrate from which the size of the sample from which the particle diameter of the crystalline cellulose shown in Table 2 differs, and microcrystal differs, the dentifrices of the following presentation were prepared and grinding force (RDA value) was measured by cleaning power and the following method.

[0025]

<Dentifrices presentation> crystalline cellulose 2% calcium hydrogen phosphate and anhydrate 30% sodium lauryl sulfate 2% Carrageenan 1% sorbitol 25% Perfume 1% purified water \*\*\*\*\* 100%[0026]The RDA (Radioactive Dentin Abrasion) value was measured by grinding force measuring method J.dent.Res., Vol.55, No.4, and the method indicated to P.563-573.

[0027]

[Table 2]

[14010 2]	サンブルNo.								
成分	11	12	13	14	15	16	17		
結晶セルロースの平均粒径 (μm)	10	180	180	70	100	180	100		
リン酸水素カルシウム・無水和 物の結晶子の大きさ(Å)	120	4200	120	400	2500	1660	1660		
清掃力評点	2	5	2	4	4.5	4,5	4,5		
RDA 値	40	240	42	90	130	115	110		
	比較品			本発明品					

[0028] From the result of Table 2, when the mean particle diameter of crystalline cellulose is [ the size of the microcrystal of 50-200 micrometers, and calcium hydrogen phosphate and anhydrate ] 300-3500A, it is accepted that a high cleaning effect is acquired by low polish nature.

[0029]

[Example 1] Tooth paste Crystalline cellulose (mean particle diameter of 120 micrometers) 5 % calcium hydrogen phosphate and anhydrate 5 (the size of microcrystal:

### 1660A)

calcium hydrogen phosphate and 2 hydrate 20 carrageenan . 0.5 Xanthan gum 0.7 propylene glycol Three Glycerin (85%) 30 sodium lauryl sulfate 2 lauric-acid decaglyceryl 0.6 sodium monofluorophosphate 0.76 Saccharin sodium 0.2 Perfume 1 total amount of residual purified water 100 %[0030]

[Example 2] Tooth paste Crystalline cellulose (mean particle diameter of 160 micrometers) 2 % calcium hydrogen phosphate and anhydrate 10 (the size of microcrystal: 2100A)

calcium hydrogen phosphate and 2 hydrate 5 silica . 10 carboxymethylcellulose sodium . 0.8 Sodium alginate 0.4 Propylene glycol Four Sorbitol (70%) 30 sodium lauryl sulfate 1.5 tranexamic acid 0.1 Saccharin sodium 0.2 Perfume 1 <u>purified water Remaining total</u> 100 %[0031]

[Example 3] Tooth paste Crystalline cellulose (mean particle diameter of 60 micrometers) 0.5 % calcium hydrogen phosphate and anhydrate 30 (the size of microcrystal: 2800A)

Calcium carbonate Five Xanthan gum One Propylene glycol 3 glycerin (85%) 15 Sorbitol (70%) 20 sodium lauryl sulfate Two Saccharin sodium 0.15 Perfume 1 <u>purified water</u> Remaining total 100 %[0032]

[Example 4] Tooth paste Crystalline cellulose (mean particle diameter of 100 micrometers) 3 % calcium hydrogen phosphate and anhydrate 7 (the size of microcrystal: 2000A)

aluminium hydroxide 25 xanthan gum . 0.5 Sodium alginate 0.5 Propylene glycol 3 sorbitol (70%) 23 sodium lauryl sulfate 1.5 Myristic acid diethanolamide 0.5 Saccharin sodium 0.2 Perfume 1 purified water Remaining total 100 %[0033]

[Example 5] Tooth paste Crystalline cellulose (mean particle diameter of 80 micrometers) 10 % calcium hydrogen phosphate and anhydrate 10 (the size of microcrystal: 2500A)

calcium hydrogen phosphate and 2 hydrate 15 silica. 3 Carrageenan 0.8 sodium alginate 0.3 Propylene glycol Three Glycerin (85%) 25 sodium lauryl sulfate 1.5 Triclosan 0.1 Saccharin sodium 0.2 Perfume 1 <u>purified water Remaining total</u> 100 %[0034] [Example 6] Tooth paste Crystalline cellulose (mean particle diameter of 180 micrometers) 15 % calcium hydrogen phosphate and anhydrate 20 (the size of

microcrystal: 500A)
Silica Five Carrageenan 0.6 Carboxymethylcellulose sodium 0.6 Propylene glyc

Silica Five Carrageenan 0.6 Carboxymethylcellulose sodium 0.6 Propylene glycol Three Sorbitol (70%) 25 sodium lauryl sulfate 1.5 Saccharin sodium 0.15 Perfume 1 <u>purified</u> water Remaining total 100 %[0035]

[Example 7] Tooth paste Crystalline cellulose (mean particle diameter of 60 micrometers) 20 % calcium hydrogen phosphate and anhydrate 25 (the size of microcrystal: 3200A)

Silica Four Carrageenan 0.3 Carboxymethylcellulose sodium 0.9 Propylene glycol Three Glycerin (85%) 30 sodium lauryl sulfate 1.5 saccharin sodium 0.2 Perfume 1 <u>purified</u> water Remaining total 100 %[0036]

[Example 8] liquefied toothbrushing Crystalline cellulose (mean particle diameter of 120 micrometers) 3 % calcium hydrogen phosphate and anhydrate 5 (the size of microcrystal: 2800A)

Silica Ten Xanthan gum 0.15. Propylene glycol Three Glycerin (85%) 45 sodium lauryl

sulfate 1.5 Lauric acid decaglyceryl 0.5 sodium monofluorophosphate 0.76 Saccharin sodium 0.2 Perfume 1 <u>purified water Remaining total</u> 100 %[0037] [Example 9] Liquefied toothbrushing Crystalline cellulose (mean particle diameter of 80 micrometers) 5 % calcium hydrogen phosphate and anhydrate 10 (the size of microcrystal: 1500A) silica 5 xanthan gum 0.2 propylene glycol . 3 Glycerin (85%) 30 sorbitol (70%) 30 sodium lauryl sulfate 1.5 Myristic acid diethanolamide 0.3 Triclosan 0.1 Saccharin sodium 0.1 Perfume 1 <u>purified water Remaining total</u> 100 %

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#### (54) 【発明の名称】 口腔用組成物

### (57)【要約】

【解決手段】 結晶セルロースパウダーを0.2~20 重量%及びX線回折法により測定した平均値が300~3500Åである結晶子を有するリン酸水素カルシウム・無水和物を含有することを特徴とする口腔用組成物。【効果】 本発明の口腔用組成物は、結晶セルロースパウダーとX線回折法により測定された平均値が300~3500Åである結晶子を有するリン酸水素カルシウム・無水和物とを併用したことにより、清掃効果が高く、低研磨性の組成物を与えるもので、商品価値の高いものである。